INVESTIGATION OF GASIFICATION OF BIOMASS IN THE PRESENCE OF CATALYSTS

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Fuels from Biomass Program

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by
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CONTENTS

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INTRODUCTION

Progress on the different project tasks that has been made since the April contractor's meeting is presented in the following discussion. Laboratory studies to determine relative catalyst activity have been completed, and work on combined catalysts is in progress. No results have been received on TGA studies from Case Western Reserve University (CWRU) but work is in progress to determine reaction kinetics. The process development unit (PDU) is installed and is currently undergoing performance evaluation tests.

The PDU will be operated to investigate methods for gasification of wood in the presence of catalysts, both single and multiple. Laboratory studies will be conducted to determine reaction kinetics and to guide the PDU studies. Feasibility studies will be conducted to determine economics of different processing schemes shown technically feasible in the PDU.

PDU installation was completed about 10 weeks late. The original schedule for milestone completion was revised to reflect this delay and still complete the study in FY-1979.

PROJECT OBJECTIVES

The overall objective of this study is to determine the technical and economic feasibility of catalyzed biomass gasification to produce specific products: a) methane, b) hydrogen, c) carbon monoxide, and d) synthesis gas for generation of ammonia, methanol, or hydrocarbons. Specific objectives are to:

1. Determine kinetics and yields from reaction between gasification media (H_2O, O_2 and/or CO_2) and wood waste with and without catalysts as a function of temperature, pressure, and gasification media.
2. Identify reaction systems from Objective 1 for production of essentially pure products (CH_4, H_2, CO and H_2/CO mixtures).
3. Design and construct a PDU for demonstration of preferred reaction systems.

4. Demonstrate preferred reaction systems in the PDU.
5. Identify and demonstrate process features required for feasibility such as:
   a) catalyst recovery and conditioning,
   b) use of ash or ash-derived material as a reaction catalyst,
   c) feed preparation, and
   d) product gas treatment.
6. Prepare conceptual designs, flowsheets, material balances, and energy balances for processes identified as technically feasible.
7. Determine economics of the technically feasible systems.

PROJECT TASKS

Four tasks to accomplish the objectives of this program were defined in the project planning document. These tasks are the following:

Task 1. Laboratory studies
Task 2. Process development unit (PDU) design, procurement, and installation
Task 3. PDU operation
Task 4. Technical and economic feasibility studies

The original and revised schedules for activities and milestones in the project tasks are shown in Figure 1. The original schedule is denoted by bars that are filled in if the activity is complete. The revised schedule is denoted by lines. Procurement delays necessitated revision of the original schedule.

Laboratory Studies

The laboratory experiments are designed to provide a sound basis for operation of the PDU. Results from these studies are intended to define the most advantageous reactant-catalyst combinations, and operating conditions. A description of the laboratory experimental system and a characterization of the various wood feed materials have been presented previously.[1,2,3] The following is a presentation of the results achieved in the laboratory task subsequent to the April Biomass Thermochemical Conversion Coordination Meeting.

Relative Catalyst Activity

The relative activity of four gasification catalysts has been examined. These catalyst are Na_2CO_3, K_2CO_3, trona ($NaHCO_3 \cdot Na_2CO_3 \cdot 2H_2O$)

CATALYTIC GASIFICATION OF BIOMASS

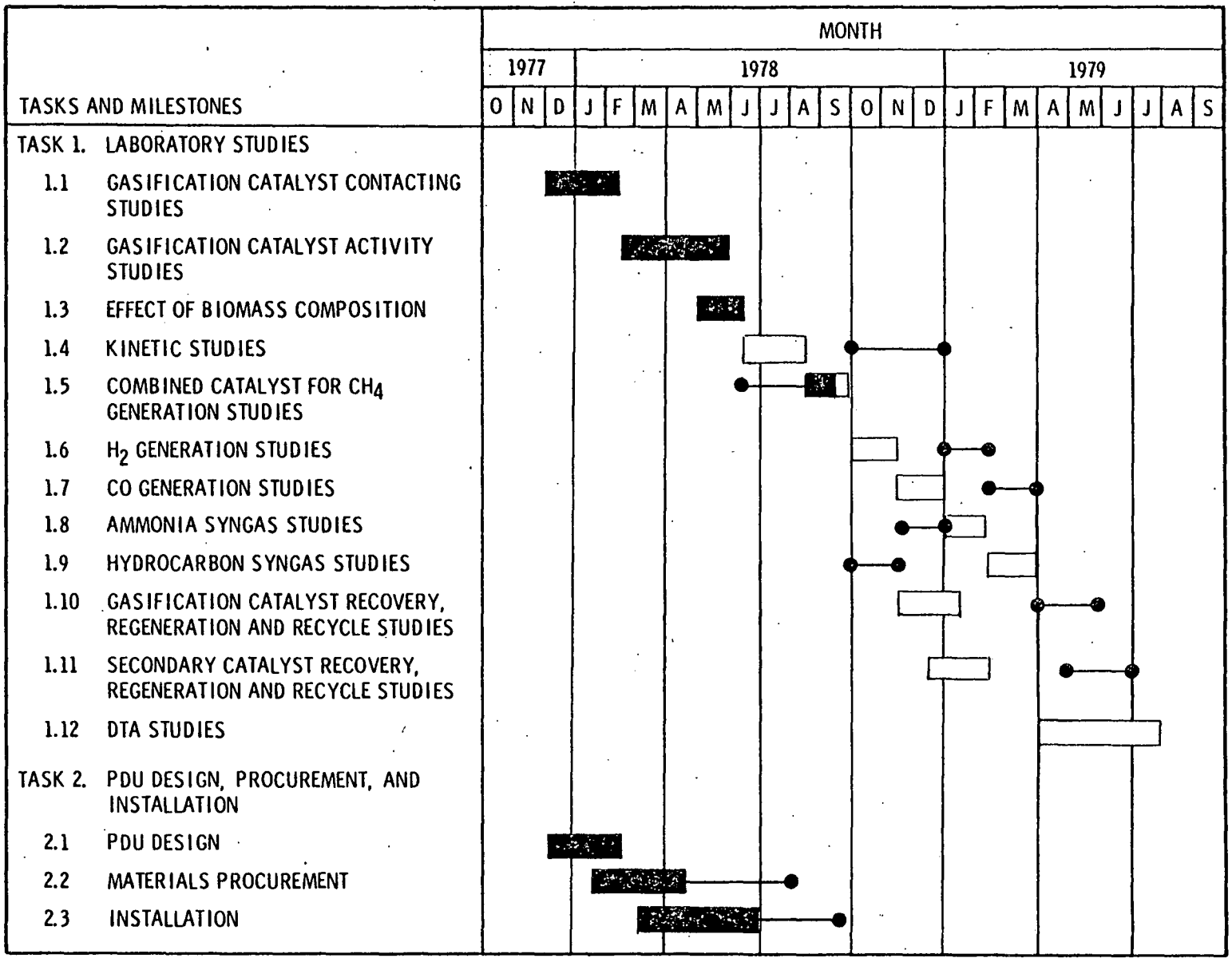

FIGURE 1. Project Schedule

CATALYTIC GASIFICATION OF BIOMASS

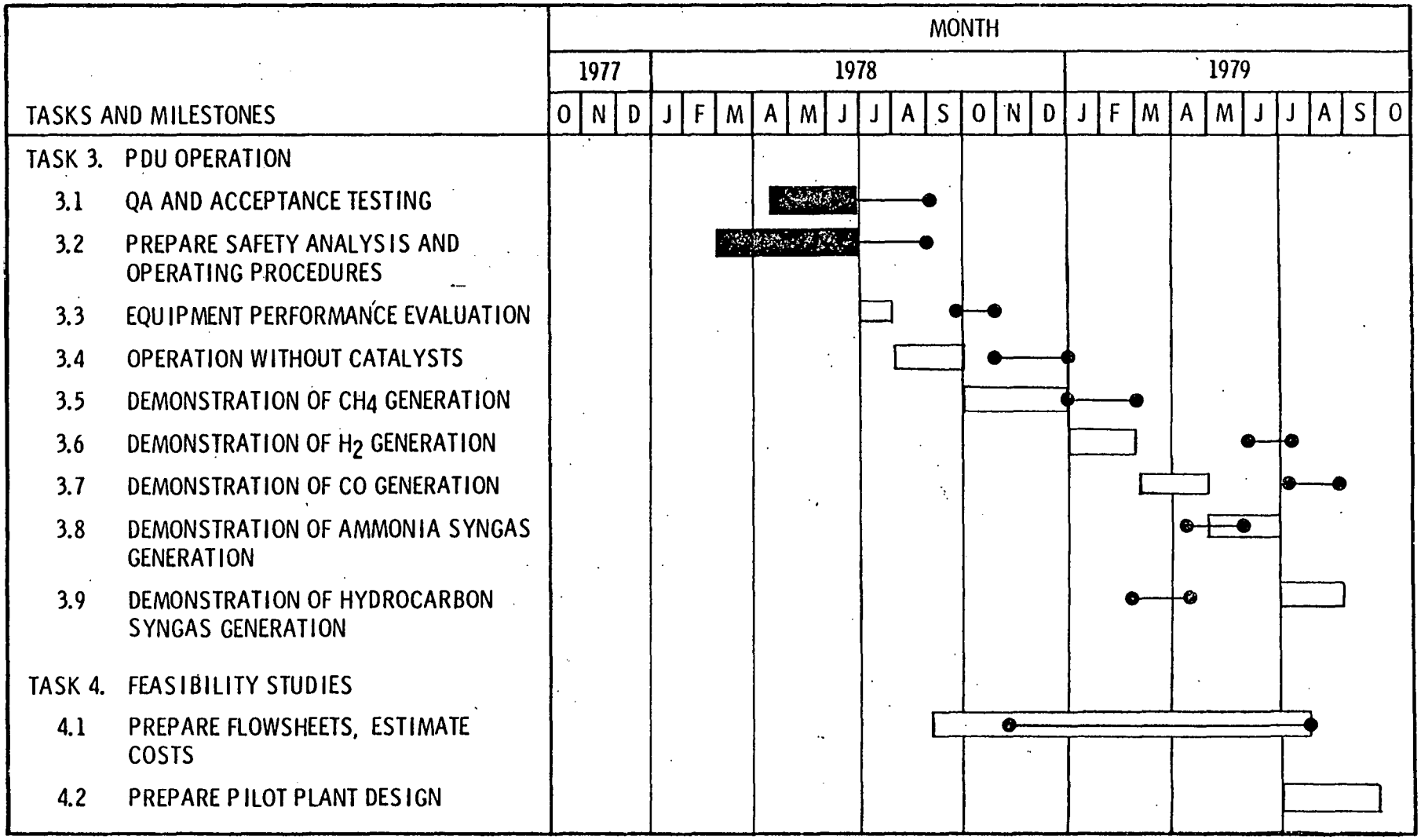

FIGURE 1. continued

MILESTONES (Figure 1, Continued)

Task 1.

1.1 Most advantageous method of contacting reference catalyst (Na_2CO_3) and biomass (wood) identified and quantified at laboratory scale, 2/16/78.

1.2 Relative activity of Na_2CO_3, trona and borax as biomass gasification catalysts established with both steam and CO_2, 6/1/78.

1.3 Relative gasification reactivity for varying moisture content and wood components (bark and heart/sap wood) established, 6/18/78.

1.4 Rate expressions determined for preferred catalyst and contacting methods over the temperature range of 400-800°C and up to 50 atmospheres, ~~8/16/78~~. 12/31/78

1.5 The variation of rate of biomass gasification (by steam) and product gas composition established at laboratory scale as functions of temperature, steam space velocity and secondary catalyst concentration to maximize CH_4 production, 10/1/78.

1.6 Variation of rate of biomass gasification (by steam) and product gas composition established at laboratory scale as functions of temperature, steam space velocity and secondary catalyst concentration to maximize H_2 production, ~~11/16/78~~. 2/14/79

1.7 Variation of CO production established at laboratory scale with biomass gasification reactant (steam with CO_2 and CO_2-O_2), temperature (650-850°C), space velocity in the presence of the preferred gasification catalyst, ~~1/1/79~~. 3/31/79

1.8 Required catalysts, operating conditions, and gasification media established at laboratory scale for production of ammonia syngas, ~~2/16/79~~. 12/31/78

1.9 Required catalysts, operating conditions, and gasification media established at laboratory scale for production of CO-H_2 syngas over the range of 1:1 to 4:1 hydrogen:carbon monoxide, ~~4/1/79~~. 11/15/78

1.10 Feasibility established and methods defined at laboratory scale for gasification catalysts recovery, regeneration, and recycling, ~~1/16/79~~. 5/15/79

1.11 Feasibility established and methods defined at laboratory scale for secondary catalyst recovery, regeneration, and recycle, ~~2/16/79~~. 6/30/79

Milestones (Figure 1. Continued)

1.12 DTA analyses completed to establish heat release/requirements for each desired product gas at optimal conditions, 8/1/79.

Task 2

2.1 PDU design completed, 2/16/78.

2.2 PDU procurement completed, ~~4/16/78.~~ 8/21/78

2.3 PDU installation completed, ~~7/1/78.~~ 9/15/78

Task 3

3.1 PDU functional/acceptance tests completed, ~~7/1/78.~~ 9/8/78

3.2 Safety approval for operation obtained. Operating procedures prepared, ~~7/1/78.~~ 8/21/78

3.3 Equipment operability established, equipment calibrated, ~~8/1/78.~~ 10/31/78

3.4 Performance without catalysts established, ~~10-1-78.~~ 12/31/78

3.5 Technical feasibility for methane generation established, ~~1/1/79.~~ 2/28/79

3.6 Technical feasibility for H_2 generation established, ~~3/1/79.~~ 7/15/79

3.7 Technical feasibility for CO generation established, ~~5/1/79.~~ 8/31/79

3.8 Technical feasibility for ammonia synthesis gas generation established, ~~7/1/79.~~ 5/31/79

3.9 Technical feasibility for hydrocarbon synthesis gas generation established, ~~9/1/79.~~ 4/15/79

Task 4.

4.1 Preliminary design for full scale systems completed. Cost estimates and economic analysis completed, 8/1/79.

4.2 Pilot plant design completed, 10/1/79.

and borax ($Na_2B_4O_7 \cdot 10H_2O$). These four inorganic compounds were chosen from the large number of possible candidates because: (1) experimental investigations using other carbonaceous materials have shown these to be the most active gasification catalysts; and (2) of the large number of possible gasification catalysts these are relatively inexpensive, e.g., Na_2CO_3 costs about $60/ton.

The relative effectiveness of the different gasification catalysts for gasification wtih steam at catalyst concentrations of 3×10^{-3} g-mol of alkali metal per g of wood is shown in Figures 2, 3 and 4 for 550°C, 650°C, and 750°C respectively. Figures 5 and 6 show the relative effectiveness of the catalyst at a concentration of 3×10^{-4} g-mol of alkali metal per gram of wood for runs at 550°C and 650°C. Results of these runs demonstrate that K_2CO_3 is by far the most effective of the gasification catalysts tested for increasing gas production rates and gas yields during steam gasification of wood. K_2CO_3 if followed by Na_2CO_3 and trona, which are about equivalent, and by Borax which exhibits very little activity in increasing rate or gas yields.

Additional results of the catalyst relative activity experiments are:

- Concentrations of 3×10^{-3} g-mol of alkali metal per gram of wood demonstrated far more catalytic activity on gasification than concentration of 3×10^{-4} g-mol of the alkali metal per gram of wood. For example, at 550°C the higher concentration of catalyst produced a two fold increase in gas production over the lower catalyst concentration.
- At catalytic concentration of 3×10^{-3} g-mol of the alkali per gram of wood K_2CO_3 produced about 1.5 times as much gas as Na_2CO_3 and trona and 3 1/2 times more gas than Borax or plain wood at both 550°C and 650°C.
- K_2CO_3 catalyzed wood produced more gas at 200 sec than is produced during the whole run for uncatalyzed wood (1500 sec) at both 550°C and 650°C.
- At 550°C K_2CO_3, Na_2CO_3, and trona catalyst concentrations of 3×10^{-4} g-mol of alkali metal per gram of wood produced very similar results. However, total gas production was about 1/2 that produced by 3×10^{-3} g-mol of K_2CO_3 at 550°C. At 650°C,

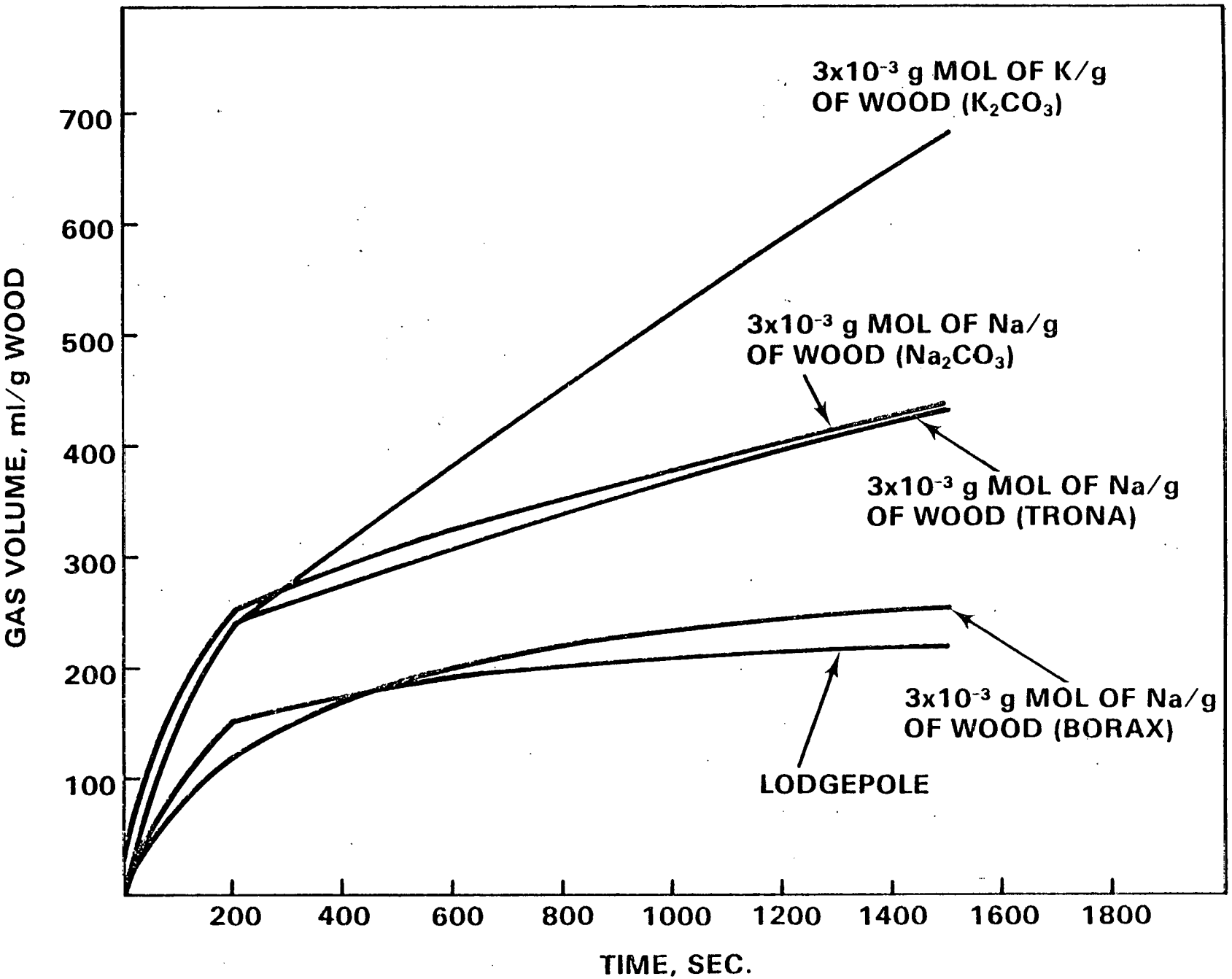

FIGURE 2 Relative Catalyst Effectiveness at 550°C for Catalyst Concentration of 3 x 10^{-3} g Mole of Alkali per g of Wood

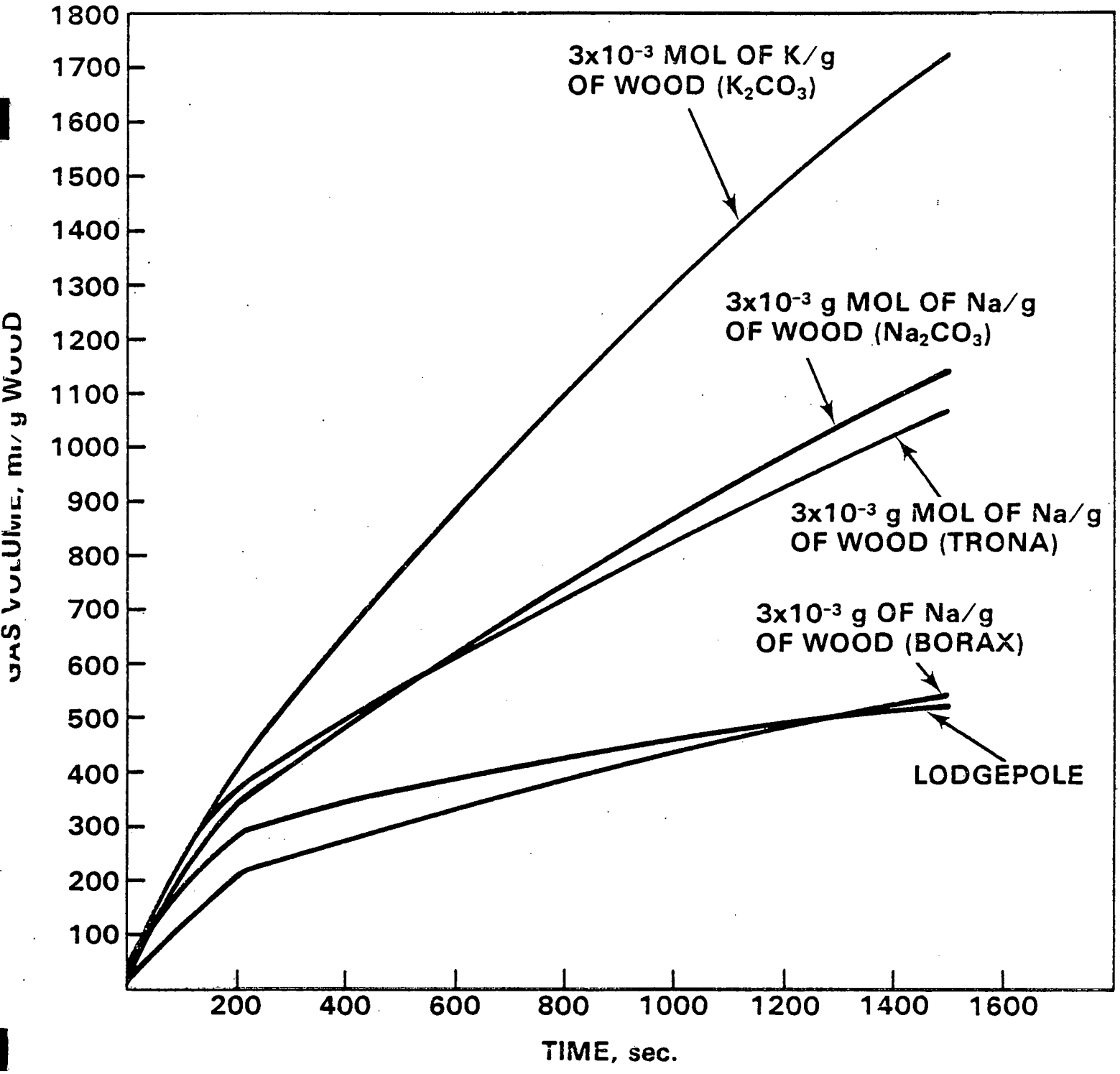

FIGURE 3 Relative Catalyst Effectiveness at 650°C for Catalyst Concentrations of 3 x 10^{-3} g Mole of Alkali per g of Wood

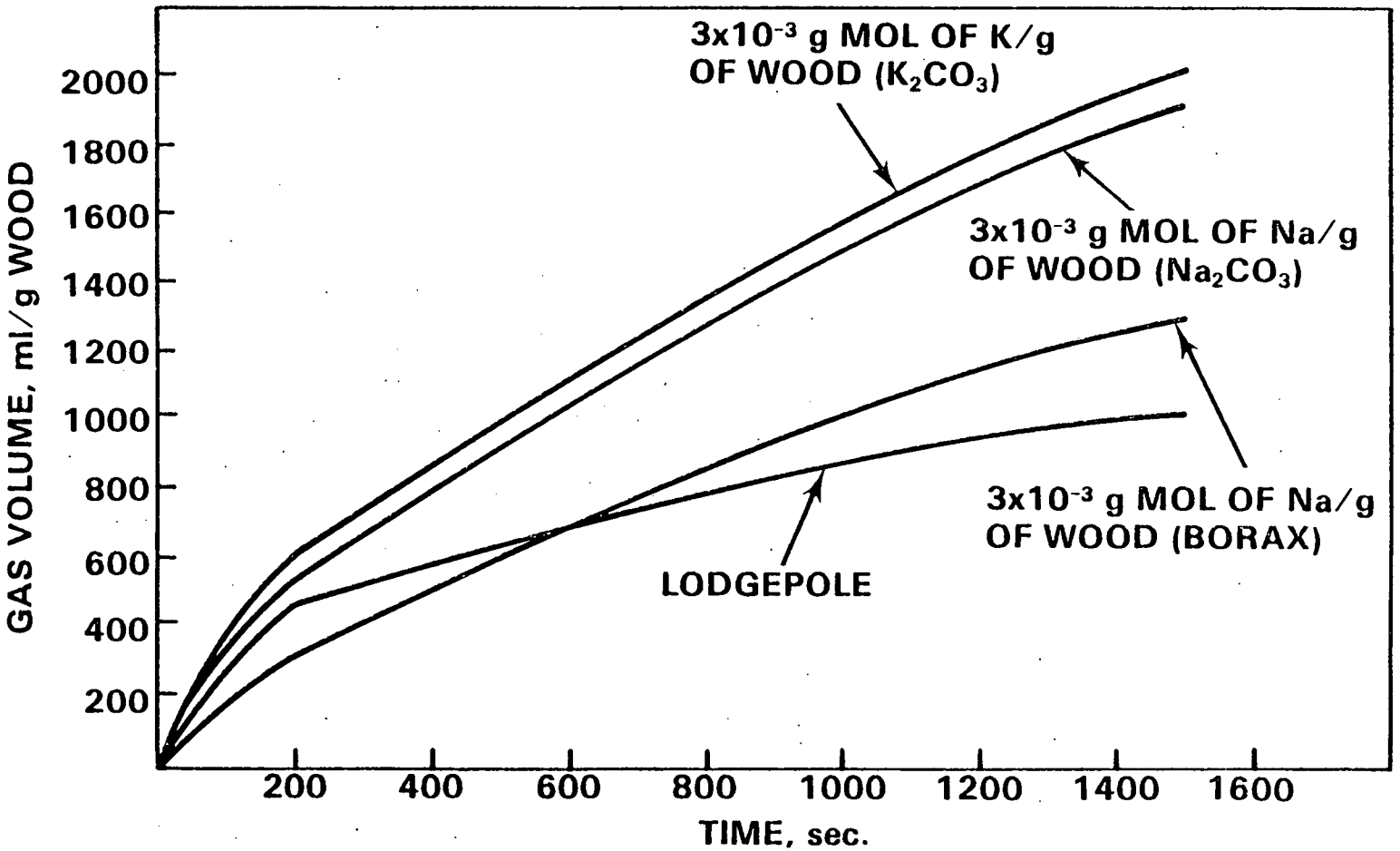

FIGURE 4 Relative Catalyst Effectiveness at 750°C for Catalyst Concentrations of 3×10^{-3} g Mole of Alkali per g of Wood

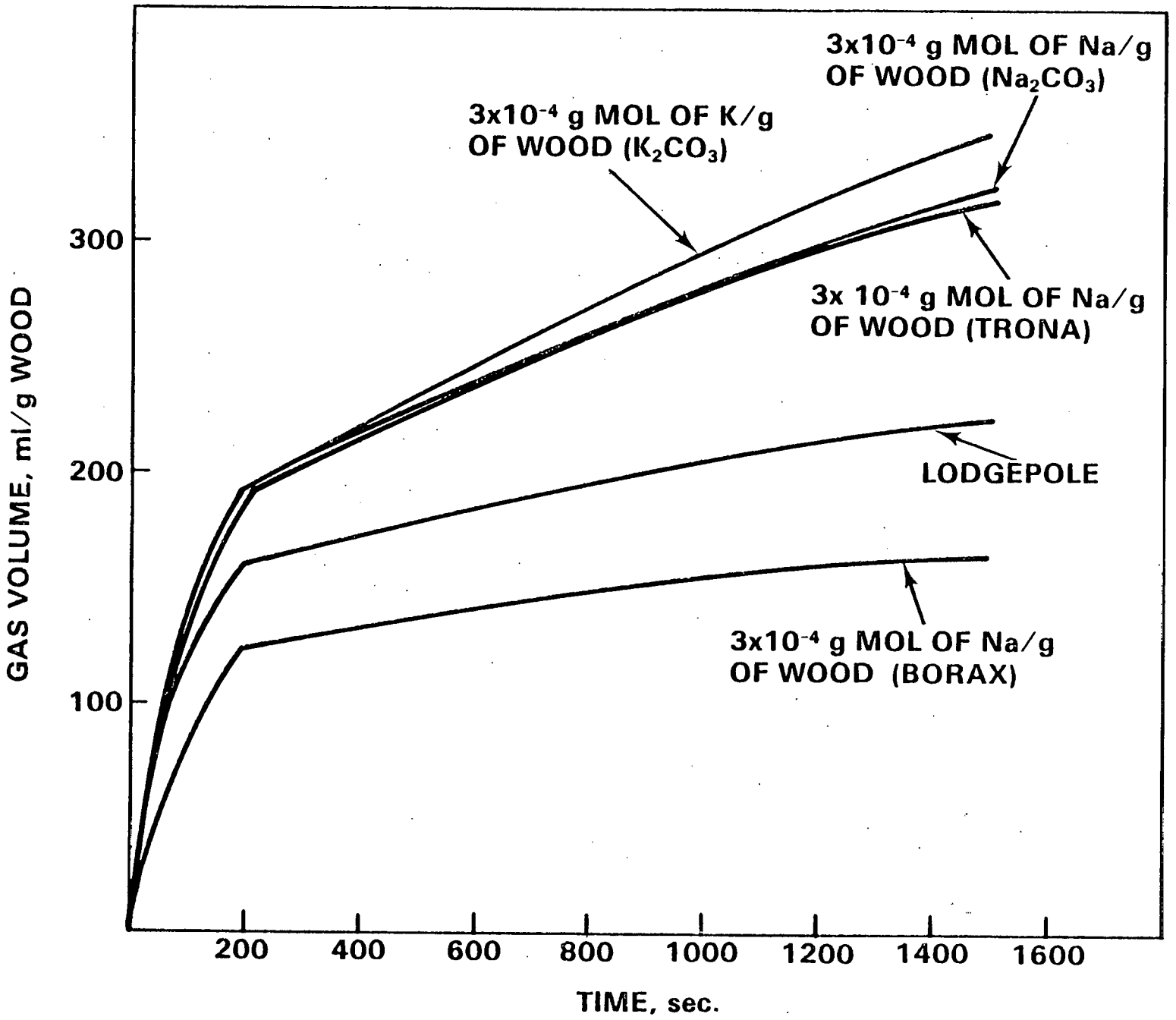

Figure 5 Relative Catalyst Effectiveness at 550°C for Catalyst Concentration of 3×10^{-4} g Mole of Alkali per G of Wood

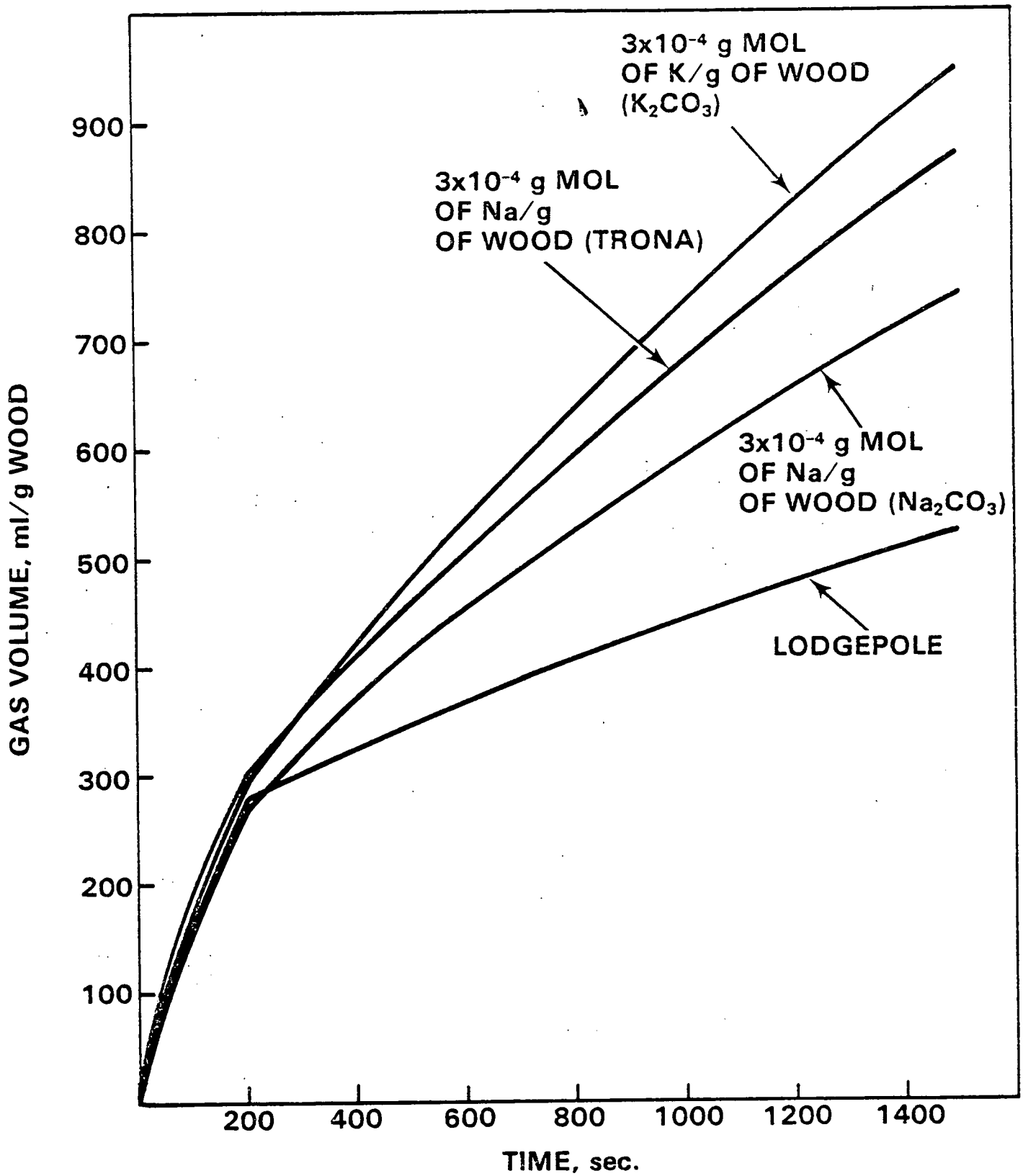

FIGURE 6 Relative Catalyst Effectiveness at 650°C for Catalyst Concentrations of 3 x 10^{-4} g Mole of Alkali per g of Wood

K_2CO_3 was about 15% better than trona and Na_2CO_3, but still less than 1/2 the gas production of runs where the higher catalyst concentrations were used.

- Gas compositions vary from one run to the next but are generally similar for K_2CO_3, Na_2CO_3 and trona. Apparent trends are hydrogen and CO_2 concentrations increase with temperature while CH_4 concentrations decrease. No difference in product selectivity was determined between K_2CO_3, Na_2CO_3 and trona during gasification of wood.

Temperature appears to be a major factor with regard to product selectivity in the absence of secondary catalyst. Increasing the gasification temperature increases the amount of hydrogen production and lowers methane and other hydrocarbon yields. Figure 7 shows the effect of temperature on gas production using a K_2CO_3 catalyst. It demonstrates that increasing the gasification temperature from 550°C to 650°C increases gas yield significantly more than an increase from 650°C to 750°C.

Effect of Biomass Composition

Four woods and bark varieties were examined to determine how gasification rates and product specificity vary with biomass composition. To accomplish this, the woods and bark as well as cellulose were impregnated with 3×10^{-3} g-mol of K per g of wood in the form of K_2CO_3 (about 20 wt. %) and gasified at 650°C. Uncatalyzed samples of each wood, bark and cellulose were gasified to serve as a data base. The results of these experiments at a gasification temperature of 650°C are:

- In all cases bark samples produced more gas than their respective woods (10-30% more in uncatalyzed runs and 10-15% more in catalyzed runs).
- In gas samples taken at 160 seconds, H_2 concentrations were higher by a factor of 4 to 6 (60% compared to 15%) for catalyzed runs for both wood and bark in comparision to their noncatalyzed counterpart. All woods showed slightly higher H_2 values and in most cases slightly lower CO and methane than their respective barks. No significant changes in product distributions or selectivity with regards to operation of the PDU are apparent at this time with respect to formation of gaseous products from the woods and barks.
- A ranking of the catalyzed woods and barks by their total gas production per gram of wood at 650°C in the presence of steam is:

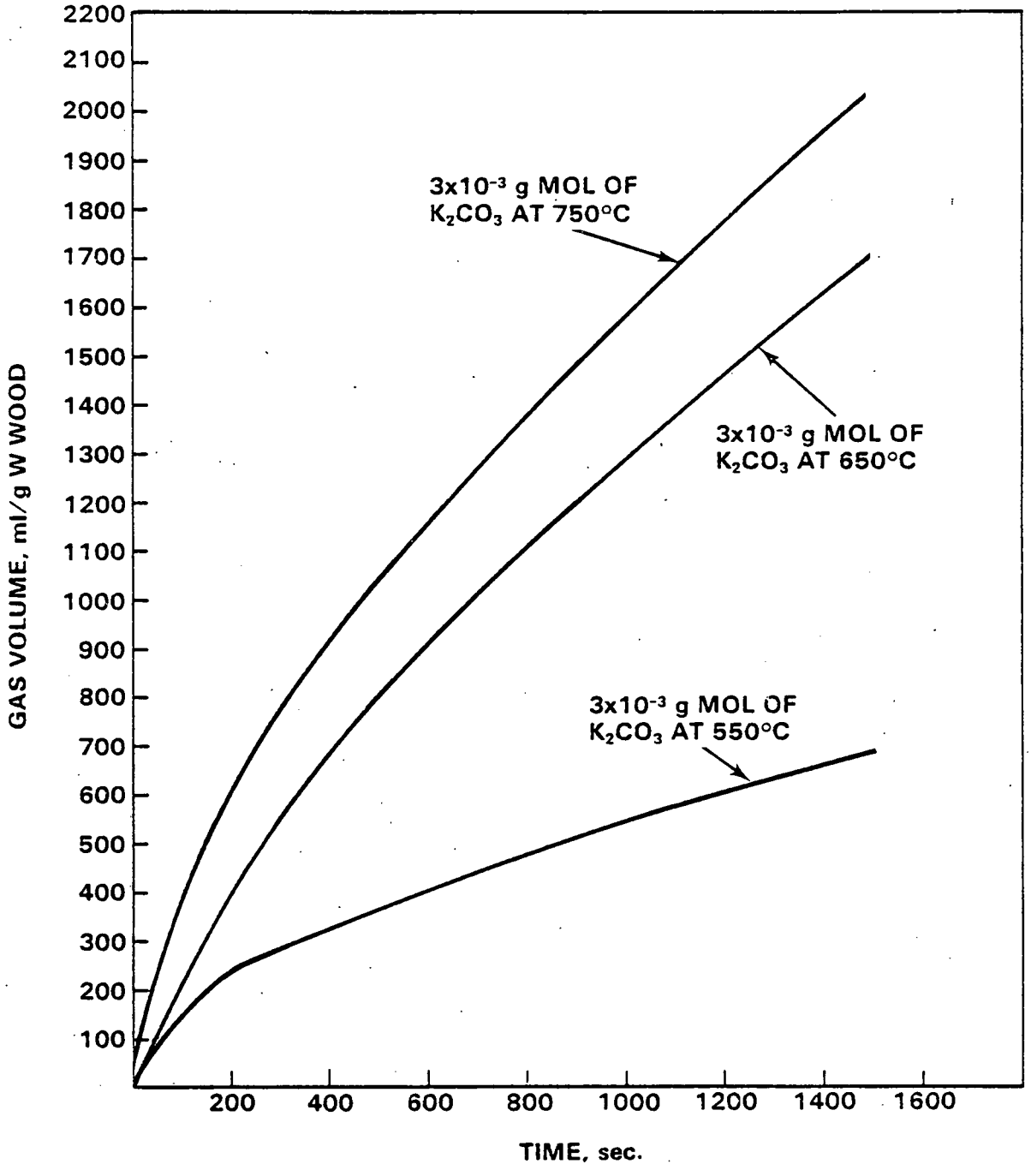

FIGURE 7 Effect of Temperature on Gas Production Using K_2CO_3 Catalyst

- Douglas Fir bark (22.1 ℓ)
- Lodgepole bark (21.4 ℓ)
- Tamarack bark (20.6 ℓ)
- Cottonwood bark (19.7 ℓ)
- Lodgepole wood (19.7 ℓ)
- Tamarack wood (19.6 ℓ)
- Douglas Fir wood (19.0 ℓ)
- Cottonwood wood (16.6 ℓ)
- Cellulose (15.3 ℓ)

- Gas yields for catalyzed runs in the presence of steam at 650°C were about 4 times greater (20 ℓ compared to 5 ℓ) for both woods and barks compared to their non-catalyzed counterparts.
- The softwood (lodgepole, tamarack, and douglas fir woods and barks) produced approximately equivalent amounts of gas at 650°C (21 ℓ for barks, 19ℓ for woods). However, cottonwood produced 1.4 times more gas uncatalyzed (7 ℓ woods, 10 ℓ bark) than the softwood wood (5 ℓ) and bark (7 ℓ), but only .9 as much as the softwood catalyzed runs (see above). This may be due to the high ash content of both cottonwood wood and bark having a catalytic effect on gasification. Gasification with wood ash as a catalyst will be tried.
- Uncatalyzed cellulose produced slightly more gas than tamarack and douglas fir uncatalyzed woods and slightly lower than cottonwood and lodgepole uncatalyzed woods. In all cases it had less gas production than uncatalyzed barks.
- In every experiment, catalyzed cellulose produced less gas than catalyzed woods and barks.
- Char residues are slightly higher for barks than wood due to their higher ash content.
- Catalyzed wood and bark samples containing up to 50% water produced equivalent amounts of total gas when gasified with steam as did dry catalyzed woods and barks. However, initial rates were somewhat slower.

Kinetics of Catalyzed Biomass Gasification

The kinetics of catalyzed biomass gasification was examined with the objective of developing a kinetic rate expression for the gasification of biomass with steam and CO_2 similar to those reported by Gardner et al[4]

and Johnson.[5] Experiments were conducted on lodgepole wood samples impregnated with about 20 wt. % K_2CO_3 to provide data to develop kinetic rate expressions for gasification with both steam and CO_2. Experiments were run at 550°, 650°, and 750°C at ambient, 10 atm, and 18 atm pressures. Preliminary results of these experiments indicate that increasing the pressure from ambient to 10 or 18 atm decreased total gas production slightly (2-5%) and had no significant effect on product distribution for steam gasification. Runs employing CO_2 as the gasifying agent demonstrate that CO_2 is poor reactant with wood at 650°C with almost total gas evolution due to devolatilization of the wood. Total gas production with CO_2 was only slightly higher than the total gas volumes produced when N_2 was the only gas present in the reactor.

Data from these experiments are being evaluated further, however it is apparent that the type of information required to generate rate expressions similar to those described above can not be achieved with current equipment available at PNL. Rate expressions will have to come from data generated by thermo gravimetric analysis (TGA) equipment at Case Western Reserve University. The results of these TGA experiments may not be applicable because they may only apply to devolatilization followed by char gasification, not the direct conversion reaction between H_2O and wood.

Combined Catalyst Performance

Experiments have been initiated to investigate the use of secondary catalysts in combination with K_2CO_3 catalysts for producing or increasing the yield of specific products. By using the proper catalyst combination and operating conditions, it is postulated that the products formed will consist primarily of either CH_4, H_2, CO, H_2-N_2, or H_2-CO. The first primary product area to be studied in the combined catalyst performance experiments is concerned with optimization of methane yield.

Methanation studies were initiated early but emphasis has been broadened somewhat to have more application to the PDU studies. Modifications have been made on existing equipment and preliminary runs are being conducted to evaluate the performance of combined catalysts. Initial methanation catalyst screening tests are currently underway with emphasis placed on development of a consistent method for catalyst

activation and on finding catalysts that do not require pre-activation. Discussions have been held with Harshaw Catalyst Inc. regarding secondary catalysts and general catalyst startup procedures.

Experiments are currently underway to develop data regarding catalyst performance. The experiments will evaluate a number of specific commercially available methanation catalysts. Factors that are being considered include (1) pre activation requirements, (2) methanation ability, (3) effect of temperature on methanation and catalyst lifetimes, (4) effect of steam and steam rate on catalyst lifetimes, and (5) other important operation factors that arise out of PDU studies.

To accomplish the initial screening of a number of methanation catalysts, the reactor has been modified slightly so that the various catalysts can be subjected to a synthesis gas mixture of $3H_2$:CO. Various reaction parameters including steam rate, temperature, and pressure can be adjusted to acquire desired data. This system has been designed to simplify experiments and to bring about better control of conditions for indefinite periods compared to dropping wood samples into the reactor. This system allows for one day evaluation of methanation catalysts at various temperatures and steam rates. A number of catalysts have been evaluated after activation and a few catalysts have been tested without prior activation. A number of other catalysts are to be tested. Data from these tests show that an increase in steam rates causes the water gas shift reaction to occur causing high H_2 yields. High temperatures also favor H_2 production and reduce CH_4 formation.

Of the catalysts tested to date Girdler G-56B appears to be of interest because it performed fairly well unactivated and at high temperature. There are a number of other catalysts that look promising that will be evaluated.

After the most promising methanation catalysts have been selected, they will be employed in the biomass laboratory reactor in both mixed and segregated runs to determine the true benefit of the various promising secondary catalysts. It is hoped that through these experiments operating conditions such as temperature, catalyst type, catalyst concentration, and steam space velocity (concentration) can be defined for PDU operation.

Subcontract with Case Western Reserve University (CWRU)

Progress to date in the TGA work being performed by CWRU has been very slow. Serious problems have been encountered with the injection of wood into the TGA. The problem stems from the plugging of wood particles in the valve between the high pressure injector and the reactor. New wood samples of a smaller mesh size (-32 + 200) were sent to CWRU to help limit the interlocking of wood particles that has been observed with larger wood particles. This improved sample injection markedly; however, sample injection is still not satisfactory. CWRU is continuing to modify their system to see if satisfactory injection can be achieved.

The steam generation system is fully constructed and ready for operation. Runs are to be made to characterize steam flow rates as a function of various operating parameters. The TGA is currently set up to conduct gasification with CO_2. Successful runs have been reported with coal char but no runs have been accomplished with wood.

PDU Design, Procurement, and Installation

Details of PDU design were presented at the April contractor's meeting. A cross section of the reactor and an equipment flow diagram are shown in Figures 8 and 9 to review the previously presented designs.

All equipment is positioned in an open, steel beam support structure. Photos of the installation taken during construction are shown in Figures 10 through 16. The main reactor was assembled in sections to allow insertion of the insulation and refractory lining, Figure 10. The main section of the reactor is comprised of two sections which were joined together before hanging on the structure as shown in Figure 11. Figures 12 and 13 show the main reactor section being transferred and attached to its position on the support structure. The cross sectional configuration of the reactor is visible in Figure 14. Metal tabs support the 1 1/2 in. refractory and 3 in. insulation. The refractory is a castable, high alumina material, and the insulation is Fiberfrax® (37% silica, 63% alumina). Figures 15 and 16 show the PDU assembly before receipt of the gas preheater. Instrumentation, visible in Figure 15, is located primarily at ground level for ease of operation. Analytical equipment is housed indoors. Sample lines and condensers are visible in Figure 16.

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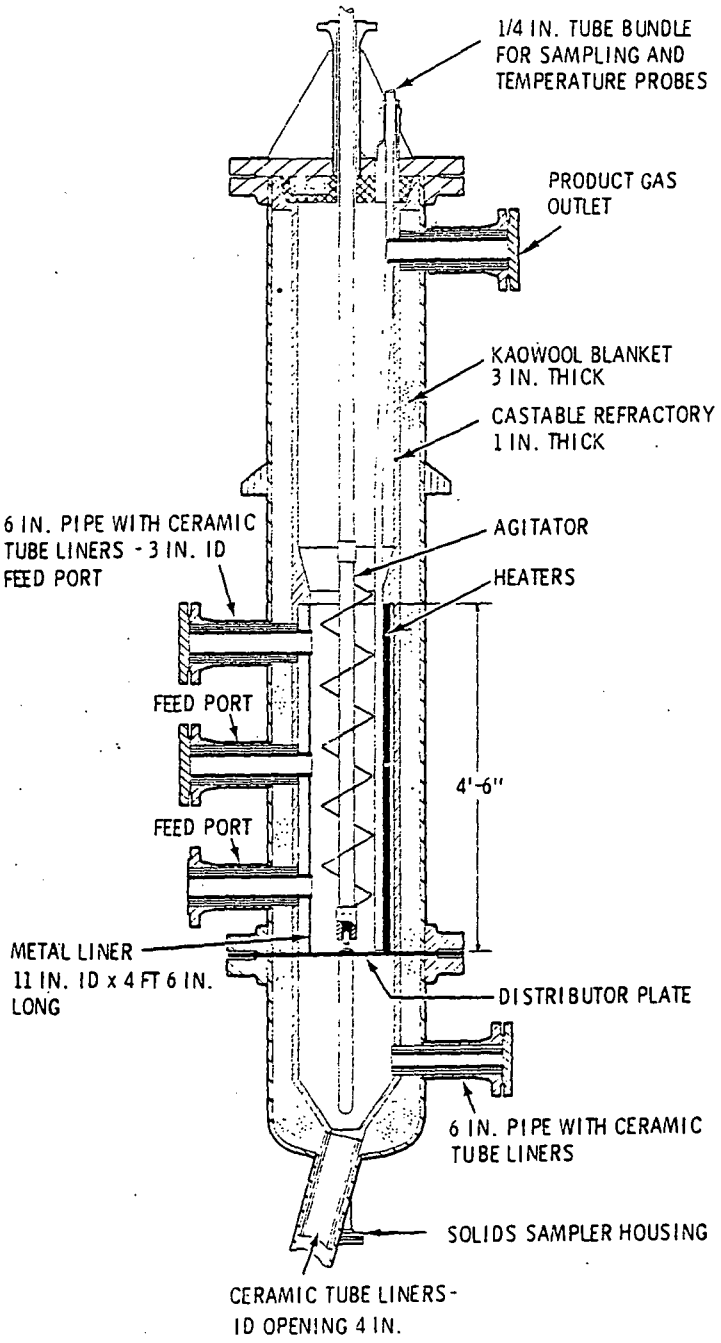

FIGURE 8 Biomass Gasification Reactor

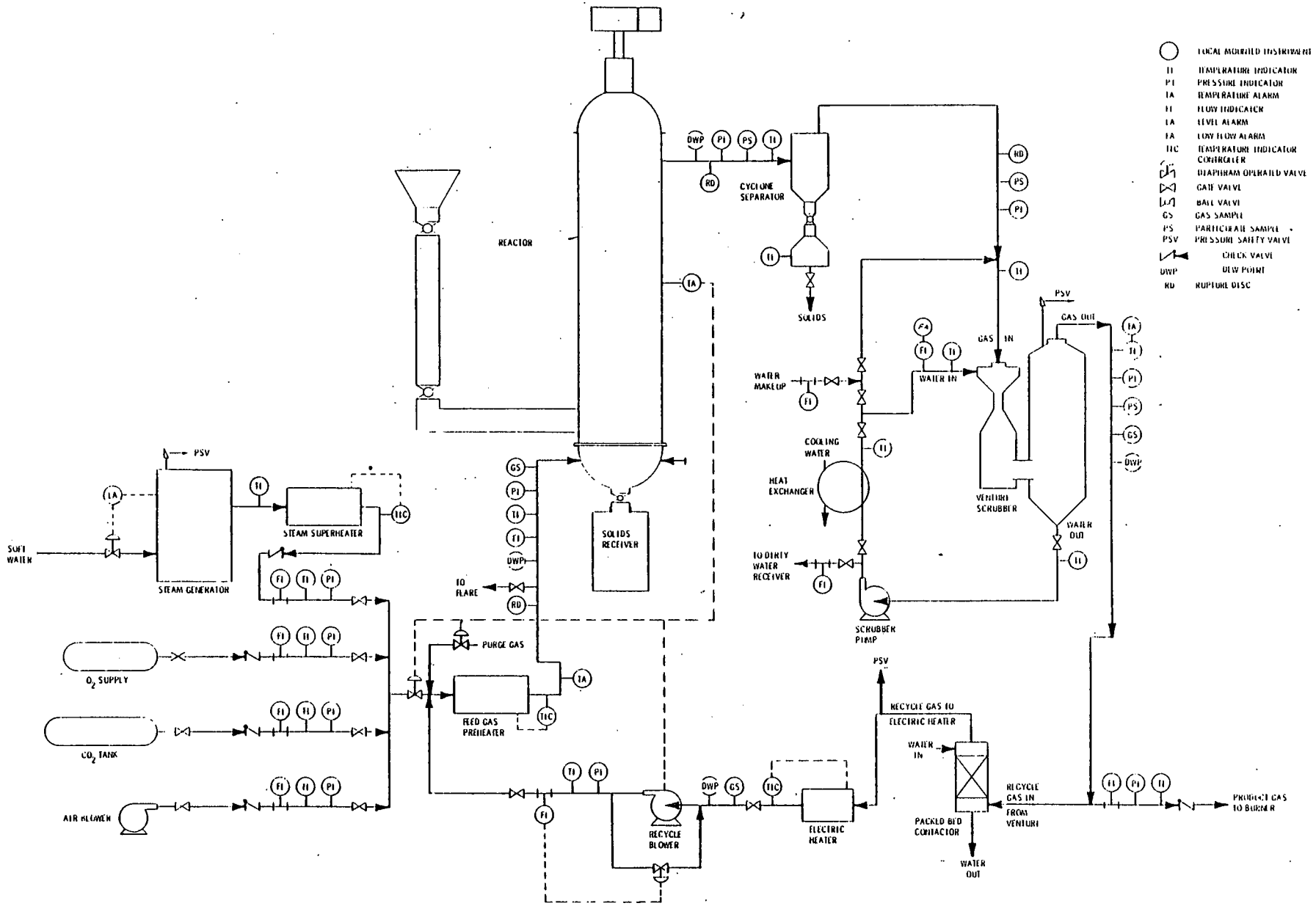

FIGURE 9 Equipment Schematic Diagram

FIGURE 10. Reactor in Sections

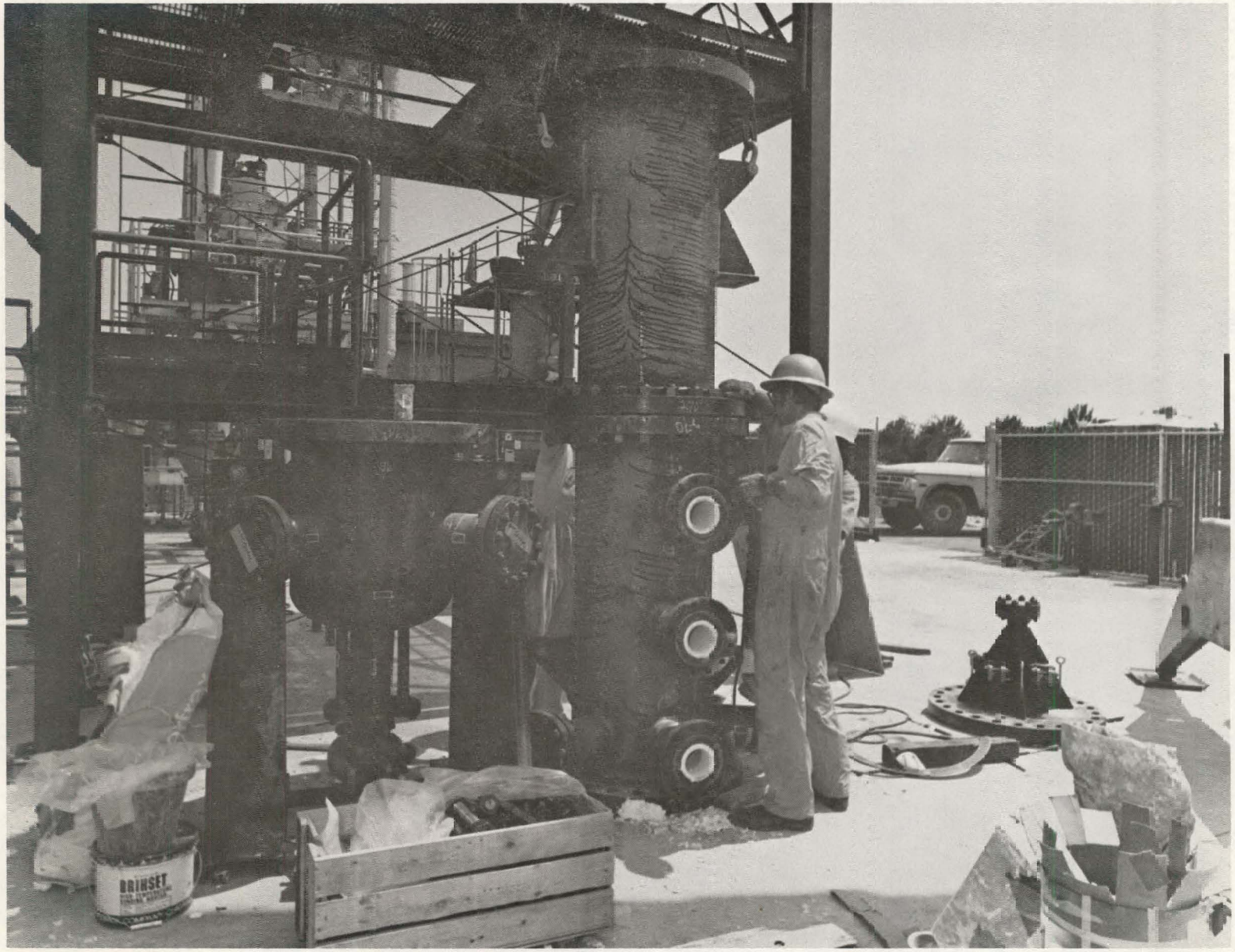

FIGURE 11. Main Section Assembled for Hanging

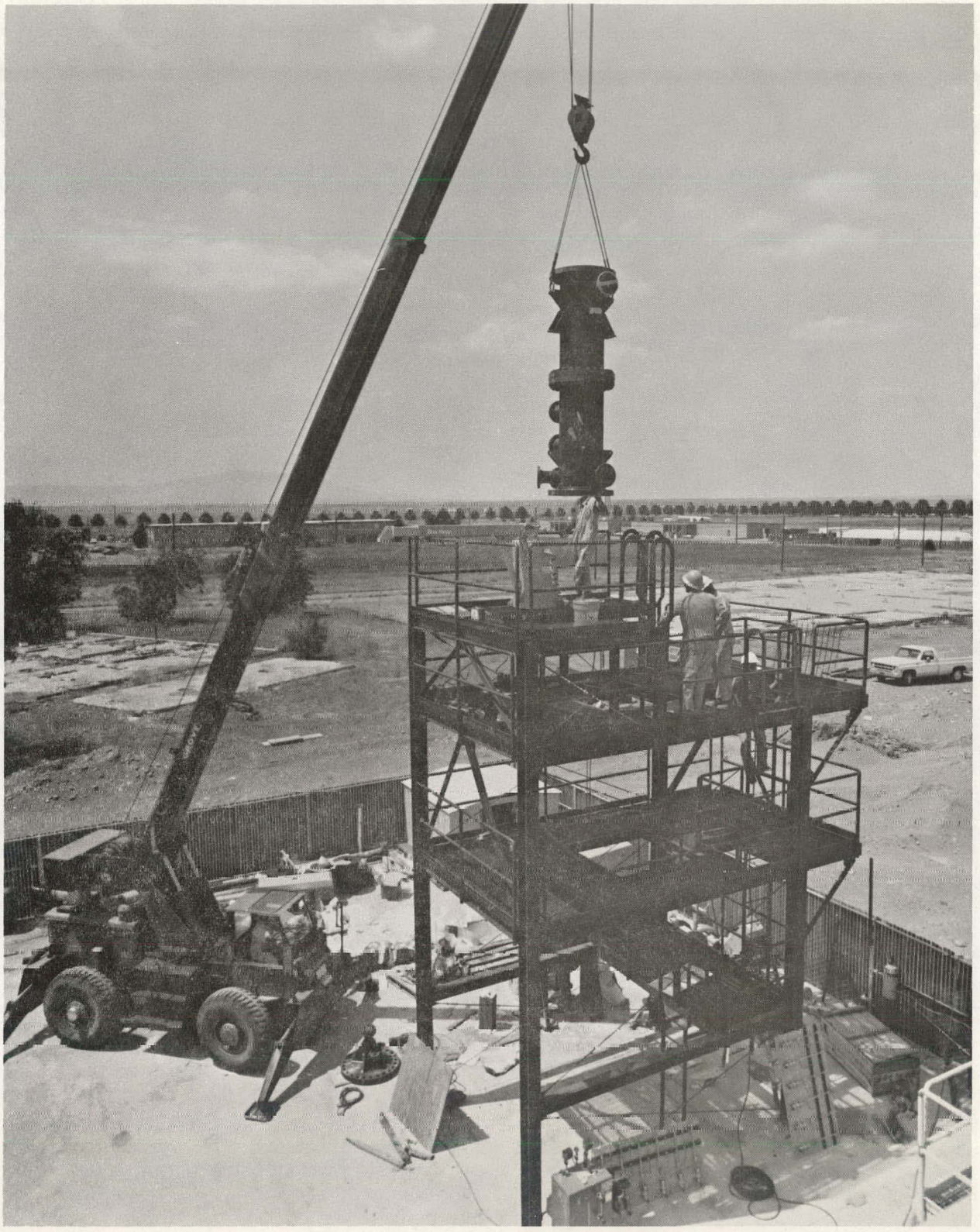

FIGURE 12. Main Section Being Transferred to Structure

FIGURE 13. Main Section In Place

FIGURE 14. View of Main Section Showing Lining and Insulation

FIGURE 15. PDU Without Gas Preheater

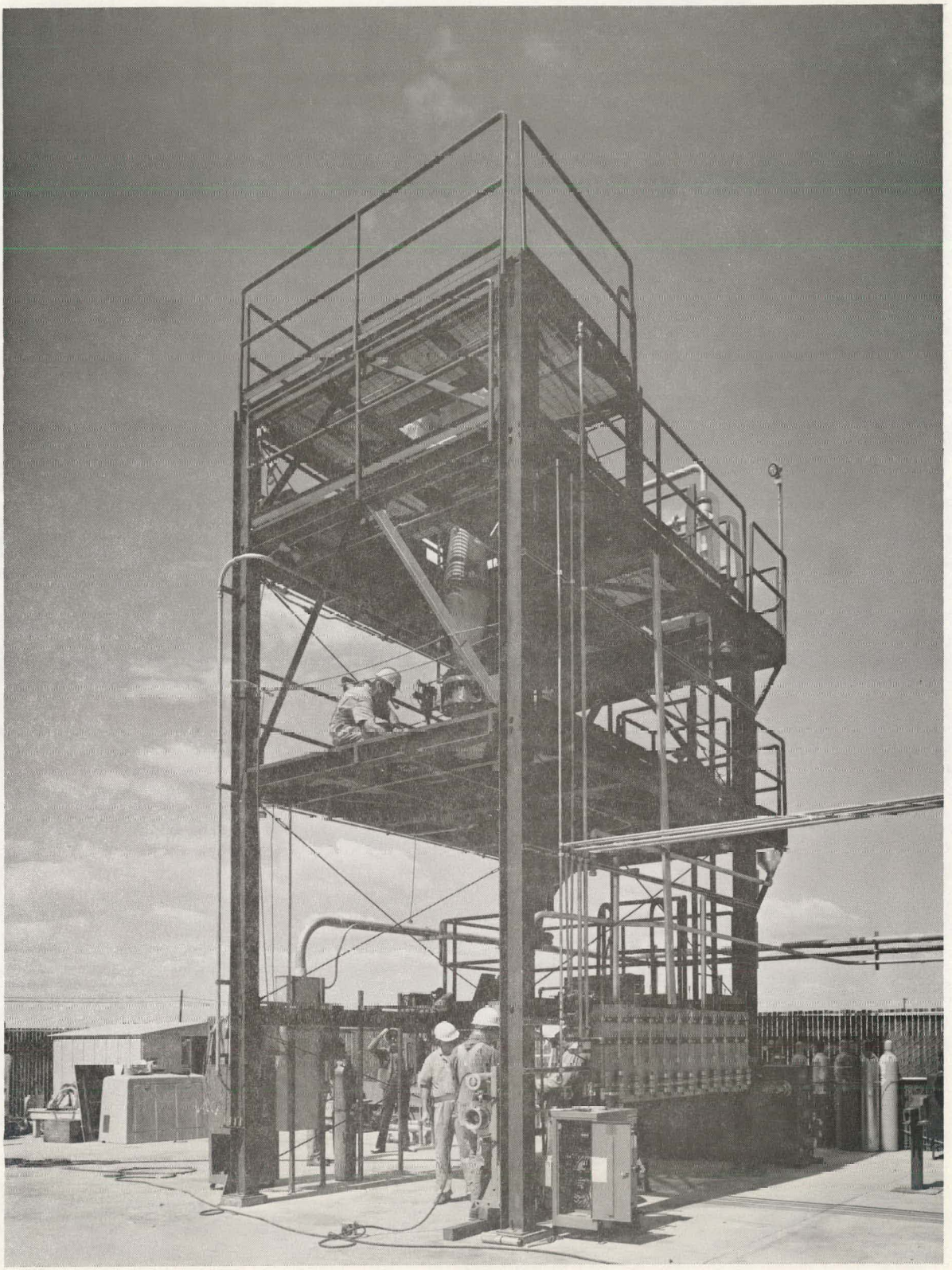

FIGURE 16. PDU View of Sample Lines

Procurement delays were the primary cause of the schedule changes shown earlier in Figure 1. Major equipment items were received about 2 months after the promised delivery date. All materials have now been received, and installation is complete. Equipment checkout is now underway. The gas preheater was used to heat air for heating the reactor to cure the refractory lining at a temperature of 550°C.

PDU Operation

Delays in completion of the PDU installation have caused similar delays in initiation of tasks on PDU operation. A safety analysis has been completed and approved by BNW safety specialists. Operating procedures have also been prepared. Quality assurance and acceptance testing of materials has been completed.

The gas preheater was used to heatup the reactor for curing of the refractory lining in the bottom section of the reactor. The gas heater performed as expected. Air temperatures up to 815°C at a flow of 50 scfm were achieved. A temperature of 800°C was reached with a 50-50 mixture of CO_2 and air at a total flow of about 75 scfm and with a mixture of CO_2 and steam.

Milestones completion dates in this task have been changed as shown in Figure 1. The PDU will be operated to answer two fundamental questions:

1) Can biomass be converted to a particular product in a single stage reactor by a judicious choice of catalysts, reactants, and reaction conditions?
2) What are the economics of the preferred process(es)? A vital part of answering this question is determining heat and material balances for the system.

The PDU runs will supply the following kinds of information needed for scale-up to a pilot plant or commercial size operation.

- Biomass sizing/feeding requirements

  Can shavings be processed as easily as sawdust? Do decomposition products depend on particle size? Will screw conveyors operate reliably at high temperatures?

- Catalyst Deactivation

  Does carbon laydown or ash deposition occur rapidly, slowly, or not at all? Will mechanical breakage (because of agitation) lower the catalyst's performance? How long will a catalyst continue to act effectively? Can it be regenerated?

Heating/Cooling Requirements

Can adding oxygen to the feed or recycle stream "burn" just enough wood to perpetuate any endothermic reactions? How can heat be removed effectively from the reactor? What is the overall heat balance for the process?

Product Gas Treatment

Will a cyclone and venturi scrubber effectively remove particulate matter from the gas stream? Can recovered catalysts be recycled directly? Do tars form which can plug downstream equipment?

Materials/Sealing Problems

Will refractory lined reactor hold up well especially with the catalyst abrasion encountered from agitation? Will the catalyst/refractory combination cause melting of the refractory? What is the corrosion rate of 316 vs 310 SS materials? Will spiral-wound gaskets adequately seal the piping or must ring joint flanges be used? Will a simple stuffing box provide an adequate seal for rotating elements?

Fundamental Information

Do kinetic expressions from laboratory studies still apply in the PDU? A reliable kinetic expression is needed to scale-up the system to pilot plant or commercial size. What is the variation of conversion with residence time? How does the selectivity ratio of a catalyst vary with time and temperature? Is isothermal operation the best or should a temperature programmed approach be used?

Answers to all of these questions are vital to a commercial plant. It is recognized that the planned 8 to 24 hour runs may not be sufficient to provide definitive answers to all of them. The quantitative information from short runs should answer most of these questions.

Feasibility Studies

Initiation of these studies has been delayed until 15 November 1978, as shown in Figure 1.

FINANCIAL STATUS

Actual and projected spending in FY 1978 is shown in Figure 17. Work for the year should be completed for the budgeted $490,000.

FIGURE 17. Project Costs

FUTURE PLANS

Laboratory studies will continue in the combined catalyst performance area over the next 8 months as shown in Figure 1. The effort will consist of maximizing the production of methane, hydrocarbon synthesis gas, ammonia synthesis gas, hydrogen, and CO respectively. This effort will be followed by studies dealing with recovery and regeneration of the alkali carbonate gasification catalyst and of the secondary catalysts.

PDU studies will be conducted as shown by the revised schedule in Figure 1. Immediate plans call for continuation of equipment performance evaluations followed by wood gasification without catalysts. Feasibility studies will start in November 1978.

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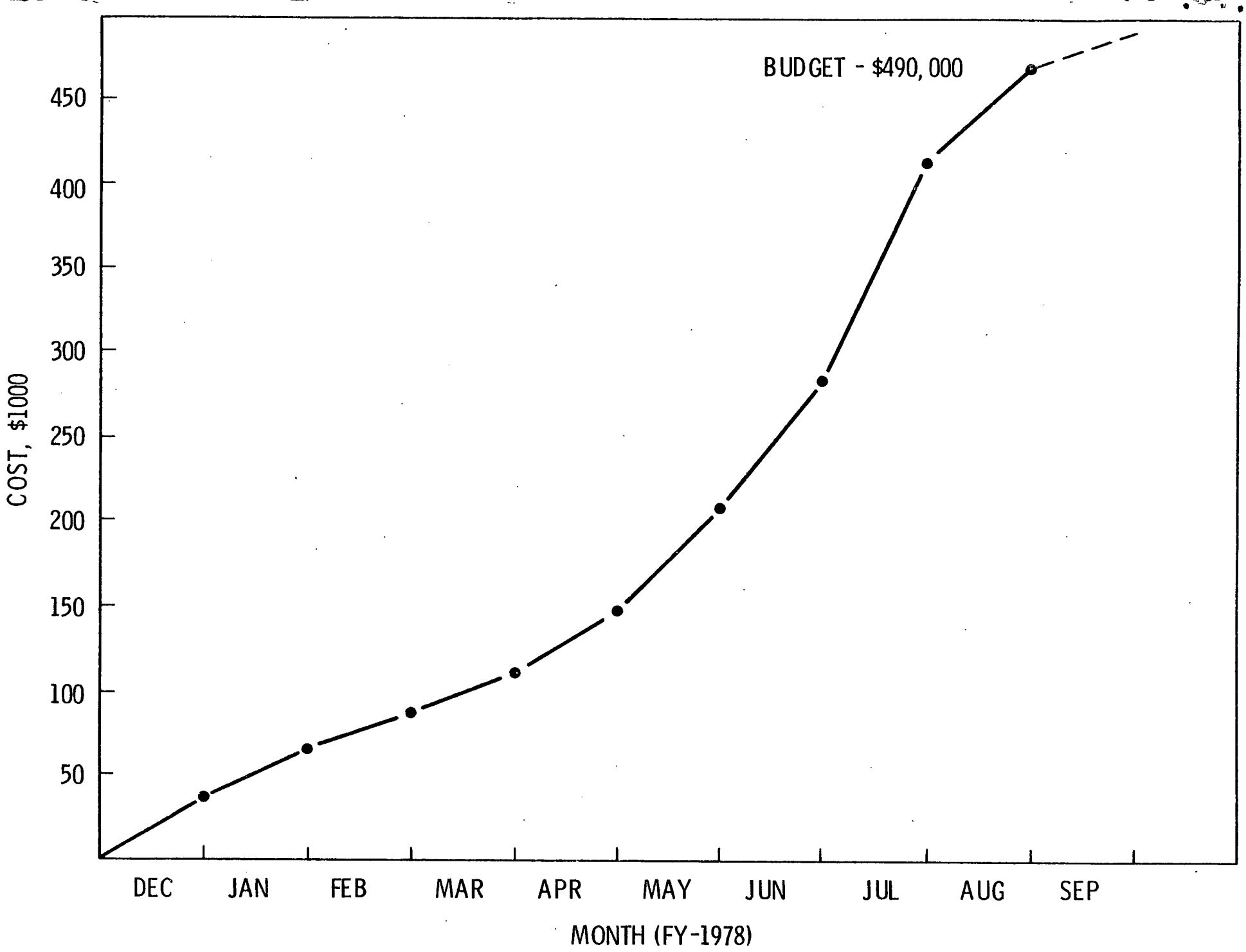

FIGURE 17. Project Costs